

**SYSTEMATICS OF PERMANENT MAGNET FILM TEXTURING  
AND THE LIMITS OF FILM SYNTHESIZED  
1-12 AND 2-17 IRON BASED RARE EARTH TRANSITION METAL  
PERMANENT MAGNET SYSTEMS**

**Final Technical Report**

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**January 21, 1998**

**U. S. Army Research Office**

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**Queens College of CUNY and Research Foundation of CUNY**

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- Fig. 2. X-Ray diffraction traces of Sm(Fe<sub>0.99</sub>Ti<sub>0.01</sub>)<sub>12</sub> films synthesized at different sputtering gas pressures of Ar are shown. The substrate temperatures were the same at 645 K.
- Fig. 3. X-Ray diffraction traces of Sm(Fe<sub>0.99</sub>Ti<sub>0.01</sub>)<sub>12</sub> films synthesized at sputtering rates are shown. The substrate temperatures were the same at 720 K, and the sputtering gas pressures were the same at 100 mTorr Ar
- Fig. 4. Hysteresis loops measured perpendicular to the film plane at 293 K, and at 10 K, are shown for (002) textured ThMn<sub>12</sub> structure Nd(Fe<sub>11</sub>Co<sub>0.5</sub>Mo<sub>0.5</sub>)N film. At 293 K: The saturation flux density was 15.5 kG. The energy product measured perpendicular to the film was 46.3 MGOe and was not coercivity limited since  $iH_c$  was 8.7 kOe. The anisotropy field estimated by extrapolating the in-plane flux density to the perpendicular value was 150 kOe. At 10 K: The saturation flux density was 17.0 kG. The coercivity was 24 kOe and the energy product measured perpendicular to the film was 59.6 MGOe. The anisotropy field estimated by extrapolating the in-plane flux density to the perpendicular value was 250 kOe.
- Fig. 5. Hysteresis loops measured at room temperatures for an ordered CaCu<sub>5</sub> phase sample with 16.7 at.% are shown for a 20 at.% Fe containing film with a predominant (110) texture are shown. This film was sputtered in 100 mTorr Ar. J. Appl. Phys. **81**, 5634 (1997).
- Fig. 6. Temperature dependence of in plane coercivity  $iH_c$ , in plane  $B_r$ , and in plane  $4\pi M$  at 18 kOe for a c-axes in plane textured SmCo based TbCu<sub>7</sub>-type film are shown as a function of temperature up to 500 °C. J. Appl. Phys. **79**, 5961 (1996).
- Fig. 7. In a subsequent paper a 7.5  $\mu$ m thick SmCo based film was deposited directly onto the Bi-YIG waveguide structure. A thin boundary layer of Al was deposited to eliminate thermal stresses. Negligible insertion losses were measured for the actual device structure. Photonics Technology Letters **8**, 903 (1996).
- Fig. 8. Room temperature hysteresis loops for a shadow deposited PLD film made from SmCo<sub>5</sub> target at 375 °C on to alumina substrate, pressure 100 mTorr Ar, pulse rate 14 Hz, are shown. The key items to note are that the intrinsic coercivity is 9.7 kOe and that the loop shape is smooth.
- Fig. 9. The coercivity  $iH_c$  versus laser pulse rate is shown for a series of films shadow deposited from SmCo<sub>5</sub> bulk targets using  $\lambda = 193$  nm, T substrate = 375 °C, and P = 100 mTorr Ar. The values shown were measured at room temperature on as deposited films.

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**Statement of Problem**

This grant was concerned with the optimization of both the magnetic properties of new rare earth transition metal permanent magnet phases, and of the film synthesis methods which have been used to synthesize high energy product film magnets and structures. High energy product permanent magnet sputtered films were synthesized onto polycrystalline substrates that either have the easy direction of magnetization in the film plane or perpendicular to the film plane. In-plane magnetized Sm-Co films were synthesized with 16-18 MGOe energy products. Other perpendicular anisotropy tetragonal Nd(Fe,Co,Mo)<sub>12</sub>N films were made with a room temperature energy product of 46.3 MGOe. The systematics of sputter process control used to grow textured permanent magnet films were elaborated in several publications. A key aspect of this work was the elucidation of surface atom mobility in controlling film texture. It was shown that the film texturing of Sm(Fe,Ti)<sub>12</sub> films could be switched between the longest stacking sequence (002) texture and the shortest stacking sequence (222) texture mode by varying individually any one of the substrate temperature, sputtering gas pressure, or sputter deposition rate. In recent studies that are still in progress pulsed laser deposition, PLD, was used to grow high coercivity SmCo based films. Room temperature coercivities up to 11.3 kOe have been achieved to date. A key aspect of this work, that also illustrates the role of surface atom mobility in growing textured magnetic films, were coercivity studies as a function of the laser pulse repetition rate. These studies showed that to obtain high coercivities, for a given substrate temperature, sufficiently low laser pulse rates were required to allow surface atom site readjustment.

## **B. Summary of the Most Important Results**

### **a. Advances in Pseudobinary Iron Based Permanent Magnets**

F. J. Cadieu, Recent Advances in Pseudobinary Iron Based Permanent Magnets, International Materials Reviews **40**, 137 (1995).

#### **Abstract**

Only recently has it been possible to make iron based analogs to Sm-Co based compounds that exhibit energy products of greater than 20 MGOe. These compounds have generally been in the form of pseudobinaries since some other element is either required to help stabilize the crystal structure or to modify the anisotropy of the base compound. The discovery of the ternary phase  $\text{Nd}_2\text{Fe}_{14}\text{B}$  was a tremendous step forward since it was the first Fe based compound that utilized the high intrinsic magnetization of Fe and large anisotropy of a rare earth element to allow very high energy density magnets to be made. But the relatively low Curie point prompted the continuing search for higher Curie point, high anisotropy, Fe based compounds. Recently five Fe based compounds have been modified through the incorporation of some third element into the structure to allow the synthesis of new high energy product, greater than 20 MGOe, Fe based magnetic materials. These systems include the compounds  $\text{Sm}_2\text{Fe}_{17}\text{N}_{3-\delta}$ ,  $\text{Sm}(\text{Fe},\text{Ti})_{12}$ ,  $\text{Nd}(\text{Fe},\text{Co},\text{Ti})_{12}\text{N}$ ,  $\text{Nd}(\text{Fe},\text{Co},\text{Mo})_{12}\text{N}$ , and  $\text{Pr}(\text{Fe},\text{Co},\text{Mo})_{12}\text{N}$ . Four of these systems use the incorporation of a small atom such as N into the structure to dramatically change the anisotropy. This review addresses some of the progress and difficulties in synthesizing these new Fe based super magnets.

## **b. High Energy Product Permanent Magnet Films**

F. J. Cadieu, High Energy Product Permanent Magnet Films, Invited Paper, in Magnetic Materials, Processes, and Devices IV, L. T. Romankiw and D. A. Herman, Jr., Editors, PV 95-18, p. 319-335, The Electrochemical Society Proceedings Series, Pennington, NJ (1996).

High energy product permanent magnet sputtered films have been synthesized onto polycrystalline substrates that either have the easy direction of magnetization in the film plane or perpendicular to the film plane. In-plane magnetized Sm-Co films have been synthesized with 16-18 MGOe energy products. Other perpendicular anisotropy tetragonal Nd(Fe,Co,Mo)<sub>12</sub>N films have been made with a room temperature energy product of 46.3 MGOe. The systematics of sputter process control used to grow textured permanent magnet films is reviewed.

### **INTRODUCTION AND BACKGROUND**

During the last few years, it has become possible to synthesize high field-strength permanent magnets in film form (1). These films can then be used to incorporate magnetic fields into the design of integrated electromagnetic components. This is a new development that allows a large number of new semiconductor, electromagnetic, and optical devices to be designed. Generally, microwave components that utilize magnetic fields have consisted of an electric circuit surrounded by a relatively large permanent magnet structure. Microwave circulators using ferrite disks and external bulk permanent magnets are an example of such devices. Magnetoresistive heads are another area in which permanent magnet films can play a significant role since magnetic field biasing is required to obtain magnetoresistive sensitivity. Small scale magnetic sensors, actuators, and motors are other areas that can benefit from the development of film permanent magnets. The magnetic field strengths that can be provided by these new film magnets are comparable to those available from bulk processed permanent magnets.

#### **Sputtering Control Parameters For Thick Film Texturing**

Three principle process control parameters can be used to synthesize preferentially textured magnetic films (13). Only one of these requires that the material being deposited be magnetic. The other two principally affect the relative probability for the growth of crystallites with various lattice parameter considerations. The three principle process considerations are:

1. the demagnetization energy required to produce a net magnetization perpendicular to the film plane versus in the film plane,
2. c/a ratio effects, and
3. the crystal structure stacking length perpendicular to substrate.

For texture control 1. to be effective requires that the anisotropy energy per grain volume be greater than  $k_B T$  at the temperature of interest. This condition is normally well satisfied for Co based hard magnetic materials, but can only be marginally satisfied for Fe based hard magnets. During the direct crystallization of films onto heated substrates, certain textures are favored from an initial random growth pattern. Once some larger seed grains become formed, there is a tendency for these grains to act as seeds for the subsequent film



growth. Techniques such as selectively thermalized sputtering allow sensitive relative growth probability factors to be propagated as the film thickness grows (14).

The following three figures show the interplay of the three principal sputtering parameters: substrate temperature, sputtering gas pressure, and deposition rate. It is shown that texture switching can be accomplished by varying any one of these three parameters independently of the others.

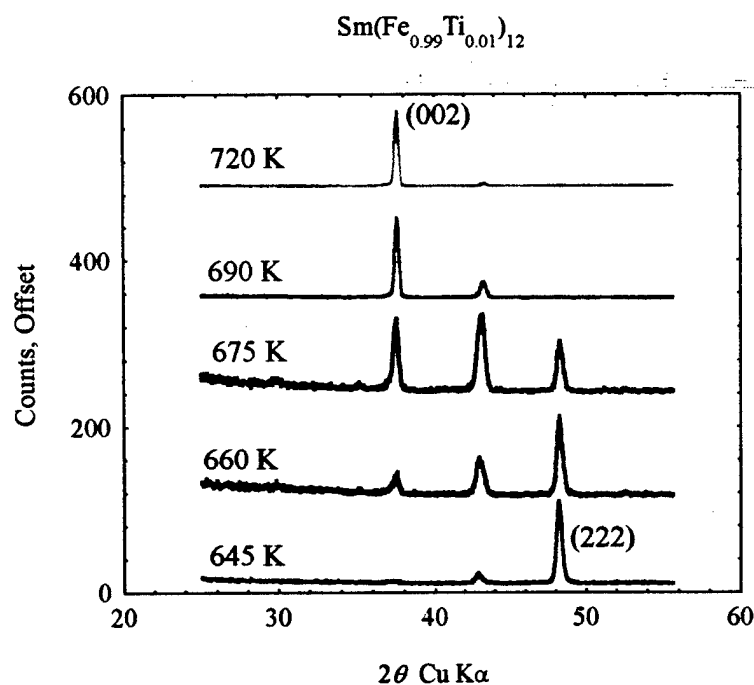


Fig. 1. X-Ray diffraction traces,  $\text{Cu K}\alpha$ , of  $\text{Sm}(\text{Fe}_{0.99}\text{Ti}_{0.01})_{12}$  films synthesized at different substrate temperatures are shown. The sputtering gas pressure was held constant at 150 mTorr Ar. The rate was 3 Å/s. The crystal structure of the films is tetragonal  $\text{ThMn}_{12}$ ,  $a = 8.50$  Å and  $c = 4.79$  Å.

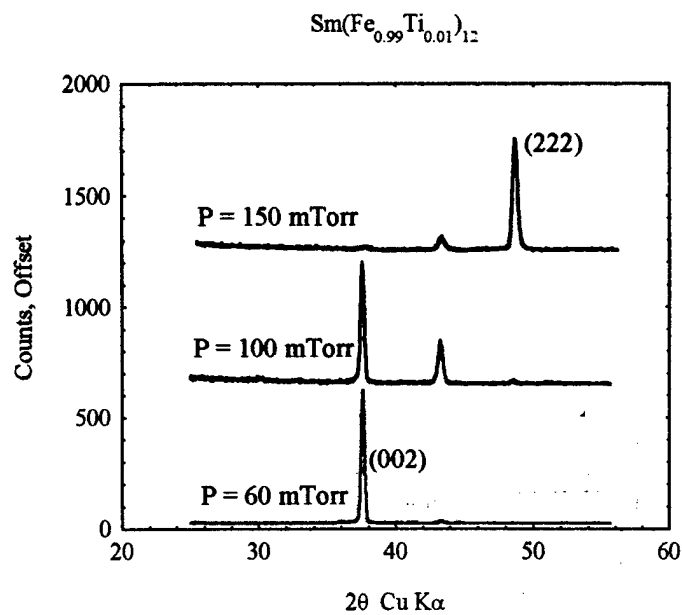


Fig. 2. X-Ray diffraction traces of  $\text{Sm}(\text{Fe}_{0.99}\text{Ti}_{0.01})_{12}$  films synthesized at different sputtering gas pressures of Ar are shown. The substrate temperatures were the same at 645 K.

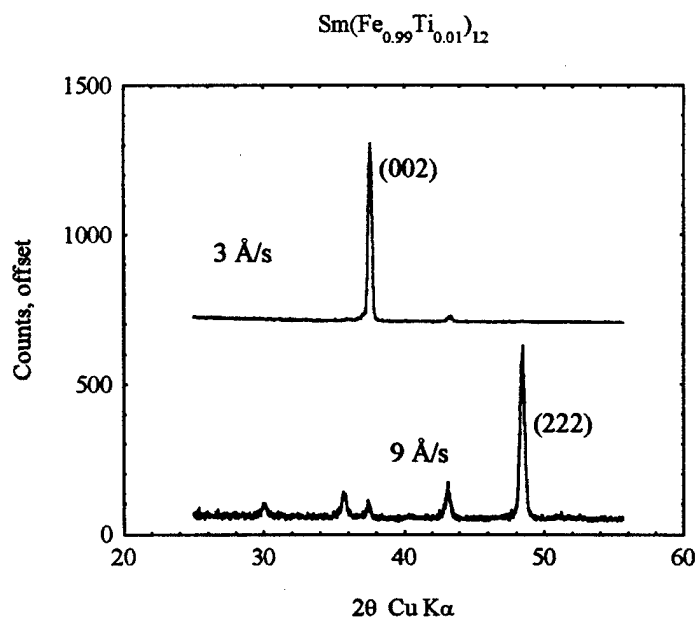


Fig. 3. X-Ray diffraction traces of  $\text{Sm}(\text{Fe}_{0.99}\text{Ti}_{0.01})_{12}$  films synthesized at sputtering rates are shown. The substrate temperatures were the same at 720 K, and the sputtering gas pressures were the same at 100 mTorr Ar.

Control of Surface Atom Mobility  
For Hard Magnet Structure Formation.

Sputtered 5  $\mu\text{m}$  Thick (002) Textured  $\text{Nd}(\text{Fe},\text{Co},\text{Mo})_{12}\text{N}$  Film

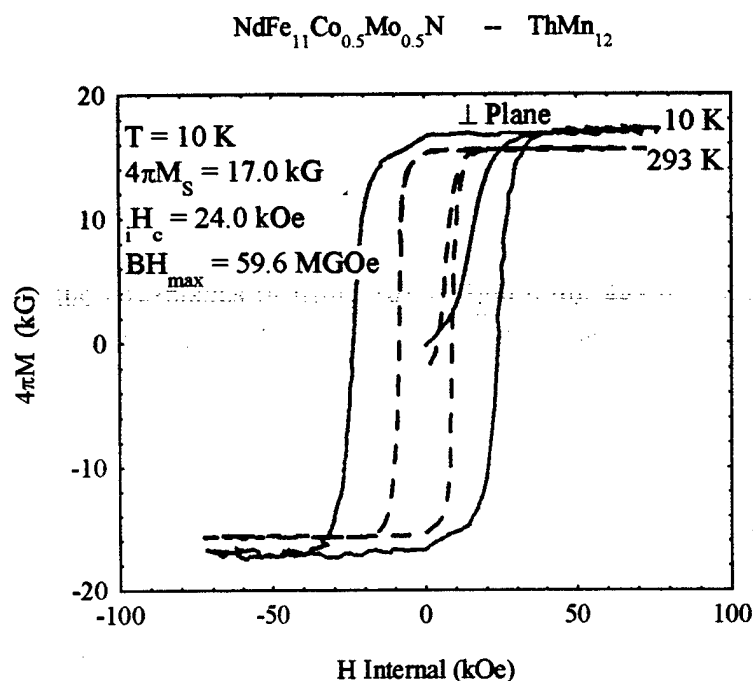


Fig. 4. Hysteresis loops measured perpendicular to the film plane at 293 K, and at 10 K, are shown for (002) textured  $\text{ThMn}_{12}$  structure  $\text{Nd}(\text{Fe}_{11}\text{Co}_{0.5}\text{Mo}_{0.5})\text{N}$  film. At 293 K: The saturation flux density was 15.5 kG. The energy product measured perpendicular to the film was 46.3 MGOe and was not coercivity limited since  $H_c$  was 8.7 kOe. The anisotropy field estimated by extrapolating the in-plane flux density to the perpendicular value was 150 kOe. At 10 K: The saturation flux density was 17.0 kG. The coercivity was 24 kOe and the energy product measured perpendicular to the film was 59.6 MGOe. The anisotropy field estimated by extrapolating the in-plane flux density to the perpendicular value was 250 kOe.

### c. The Sputter Synthesis of SmCo Based Films For Device Type Applications.

#### Sputter Synthesis of TbCu<sub>7</sub> Type Sm(CoFeCuZr) Films With Controlled Easy Axis Orientation, J. Appl. Phys. **76**, 6760 (1994).

This paper dealt with the synthesis of highly textured films of SmCo based films with the crystallite c-axes aligned onto the film plane. Such films exhibit an extreme degree of in-plane anisotropy. Typically such films optimally prepared have remanent flux densities of 8-8.5 kG, intrinsic coercivities of 6-8 kOe, and room temperature in-plane static energy products of 16-19 MGOe. The Curie temperature of this system is approximately 700 °C so that such films are useful to relatively high temperatures. One of the results of this paper is the advantages of using Ar-Xe sputtering gas mixtures to maximize the degree of in-plane anisotropy and the remanent magnetization.

Such films have been used to fabricate several types of small scale device geometries. Films with thicknesses up to 120 μm have been deposited onto polycrystalline aluminum oxide, Si, and GaAs substrates. Boundary layers have been necessary to obtain film to substrate adhesion for films thicker than about 12 μm.

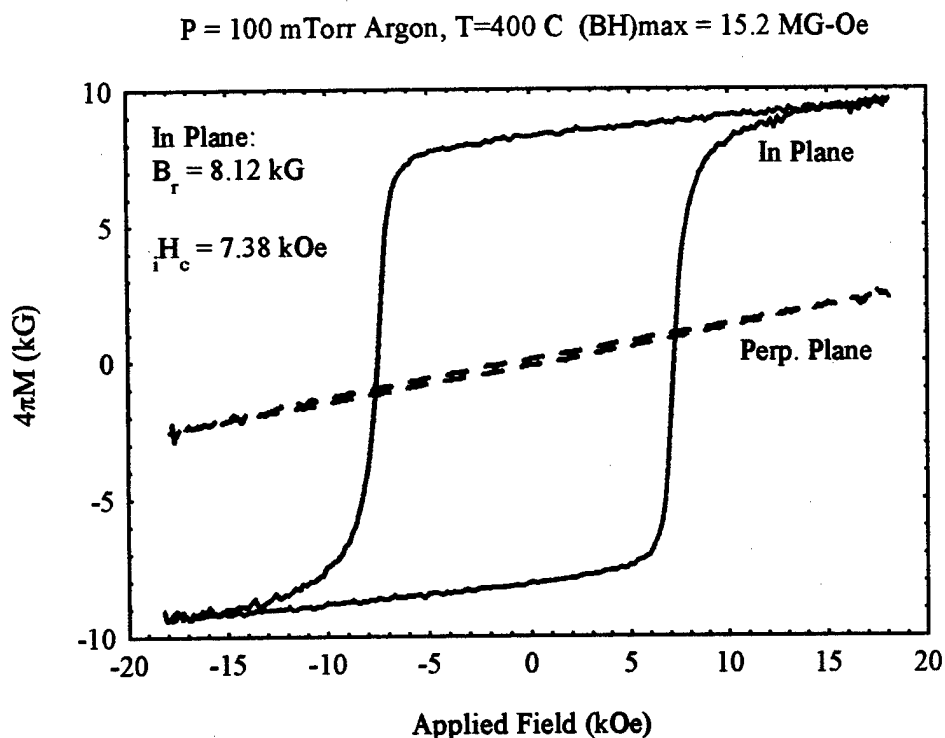


Fig. 5. Hysteresis loops measured at room temperatures for an ordered CaCu<sub>5</sub> phase sample with 16.7 at.% are shown for a 20 at.% Fe containing film with a predominant (110) texture are shown. This film was sputtered in 100 mTorr Ar. J. Appl. Phys. **81**, 5634 (1997).

#### **d. High Temperature Properties of TbCu<sub>7</sub>-type SmCo Based Films**

H. Hegde, X. R. Qian, Jong-Guk Ahn, and F. J. Cadieu,  
High Temperature Magnetic Properties of TbCu<sub>7</sub>-type SmCo Based Films, Paper FF-09, 40th Magnetism and Magnetic Materials Conference, Philadelphia, Nov. 6-9, 1995, J. Appl. Phys. **79**, 5961 (1996).

Work on TbCu<sub>7</sub>-type SmCo based films has been continued under U. S. Army Research Office support. The high temperature magnetic properties of this system have been prepared for presentation at the 40th MMM Conference to be held in Philadelphia, November 6-9, 1995. Normally bulk samples in this composition range form the two phase cellular structure whose magnetic properties are very sensitive to heat treatments. Single phase TbCu<sub>7</sub>-type films of composition Sm<sub>13</sub>Co<sub>58</sub>Fe<sub>20</sub>Cu<sub>7</sub>Zr<sub>2</sub> were sputter synthesized such that the crystallite c-axes were oriented in the film plane. Optimal magnetic properties are obtained from the as sputtered films. The magnetic properties of these single phase films are insensitive to subsequent thermal heat treatments. The net anisotropy field of the films remained larger than the maximum applied field of 18 kOe even at the highest measurement temperature of 460 °C. Thus all measurements were from minor hysteresis loops. The in plane coercivity showed a monotonic decrease from 6.0 kOe at room temperature to 1.3 kOe at 460 °C. The in plane remanent flux density decreased from 8.5 kG at 50 °C to 5.8 kG at 460 °C.

The coercivity, remanent induction, and flux density at an applied field of 18 kOe for a SmCo based TbCu<sub>7</sub> type film as a function of temperature are shown in the figure below.

# Sm-Co Based TbCu<sub>7</sub>

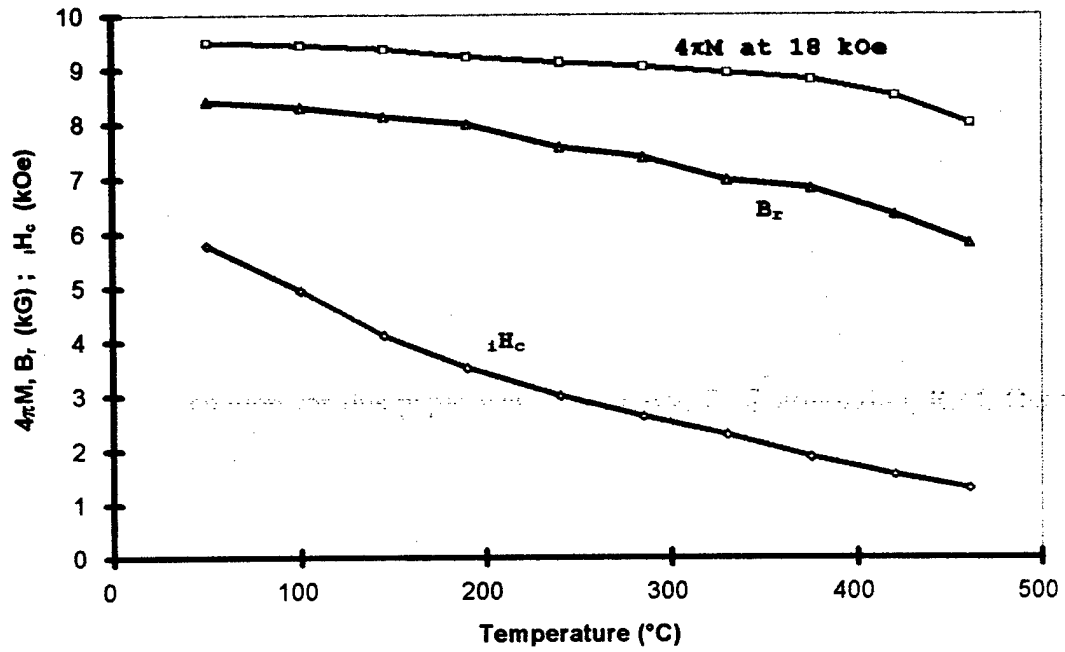


Fig. 6. Temperature dependence of in plane coercivity  $H_c$ , in plane  $B_r$ , and in plane  $4\pi M$  at 18 kOe for a c-axes in plane textured SmCo based TbCu<sub>7</sub>-type film are shown as a function of temperature up to 500 °C. J. Appl. Phys. **79**, 5961 (1996).

### **e. PERMANENT MAGNET FILM MAGNETOOPTIC WAVEGUIDE ISOLATOR**

In Conjunction With: Columbia University Microelectronics Sciences Laboratory,  
AT&T Bell Laboratories, and the Naval Research Laboratory

As a part of this research a film based high performance magnetooptic waveguide isolator has been constructed and tested. In a preliminary device configuration consisting of a waveguide etched into a Bi-YIG film, a SmCo based permanent magnet film deposited onto a separate substrate was used to saturate the magnetization of the Bi-YIG waveguide. The TbCu<sub>7</sub> SmCo based film magnet was 22  $\mu\text{m}$  thick in this case. A 45° rotation for a 1550 nm light wavelength was provided by a Bi-YIG light-pipe length of 3.55 mm with measured isolation ratios of 25 dB for light wavelengths from 1490 to 1555 nm. The publication citation for this paper was -- M. Levy, R. Scarmozzino, R.M. Osgood, F.J. Cadieu, and H. Hegde, "Permanent Magnet Film Magnetooptic Waveguide Isolator", Paper DP-27 38th MMM, Minneapolis, November 15-18, 1993, J. Appl. Phys. **75**, 6286 (1994).

In the following paper a 7.5  $\mu\text{m}$  thick SmCo film with suitable boundary layers was deposited directly onto the Bi-YIG waveguide. This was accomplished without any additional insertion losses. The isolation ratio was 28-30 dB over the light wavelength of 1490 to 1555 nm. This paper was published in Photonics Technology Letters -- M. Levy, R. M. Osgood, Jr., H. Hegde, F. J. Cadieu, R. Wolfe, and V. J. Fratello, "Integrated Optical Isolators With Sputter-Deposited Thin-Film Magnets", Photonics Technology Letters **8**, 903 (1996).

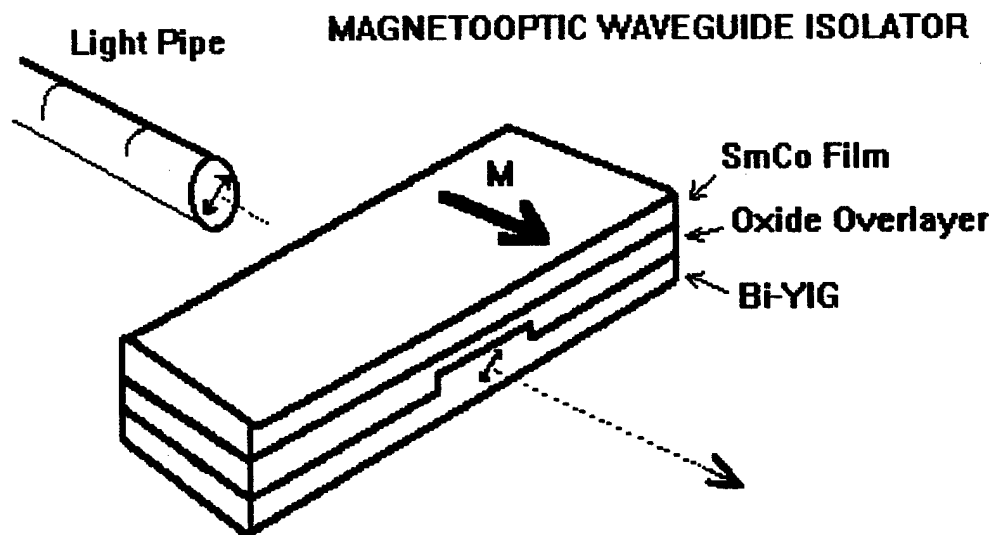


Fig. 7. In a subsequent paper a 7.5  $\mu\text{m}$  thick SmCo based film was deposited directly onto the Bi-YIG waveguide structure. A thin boundary layer of Al was deposited to eliminate thermal stresses. Negligible insertion losses were measured for the actual device structure. Photonics Technology Letters **8**, 903 (1996).

## **f. Initial Results on Permanent Magnet Films by Pulsed Laser Deposition**

Paper AD-04 7th Joint MMM-INTERMAG, San Francisco, Jan. 1998  
Accepted For Publication, Journal of Applied Physics

### **High Coercivity SmCo Based Films Made by Pulsed Laser Deposition**

F. J. Cadieu, R. Rani, X. R. Qian, and Li Chen  
Physics Department, Queens College of CUNY, Flushing, NY 11367

#### **Abstract**

Films of SmCo based materials exhibiting high intrinsic coercivities and smooth hysteresis loops have been prepared by pulsed laser deposition, PLD, onto moderately heated substrates. Films directly crystallized from SmCo<sub>5</sub> targets onto 375 °C substrates exhibited a maximum  $\mu_0 H_c = 11.3$  kOe at a pulse repetition rate of 10 Hz with lower coercivities for both lower and higher pulse repetition rates. In the present case the films were deposited onto polycrystalline substrates. The films exhibited a very small grain size of less than 1  $\mu\text{m}$  diameter, were mirror-like, and shadow deposited films were relatively particulate free under SEM examination. Shadowed PLD deposition was used for the best films. Laser wavelengths of 193 and 248 nm were used with pulse repetition rates from 5 to 50 Hz. Films grown without shadowing exhibited a great deal of particulate contamination. The hysteresis loops of such non-shadowed films were constricted and exhibited drops in the  $4\pi M$  values upon demagnetization. To our knowledge this is the first reporting of high coercive force SmCo based films deposited by PLD exhibiting single phase type hysteresis loops.



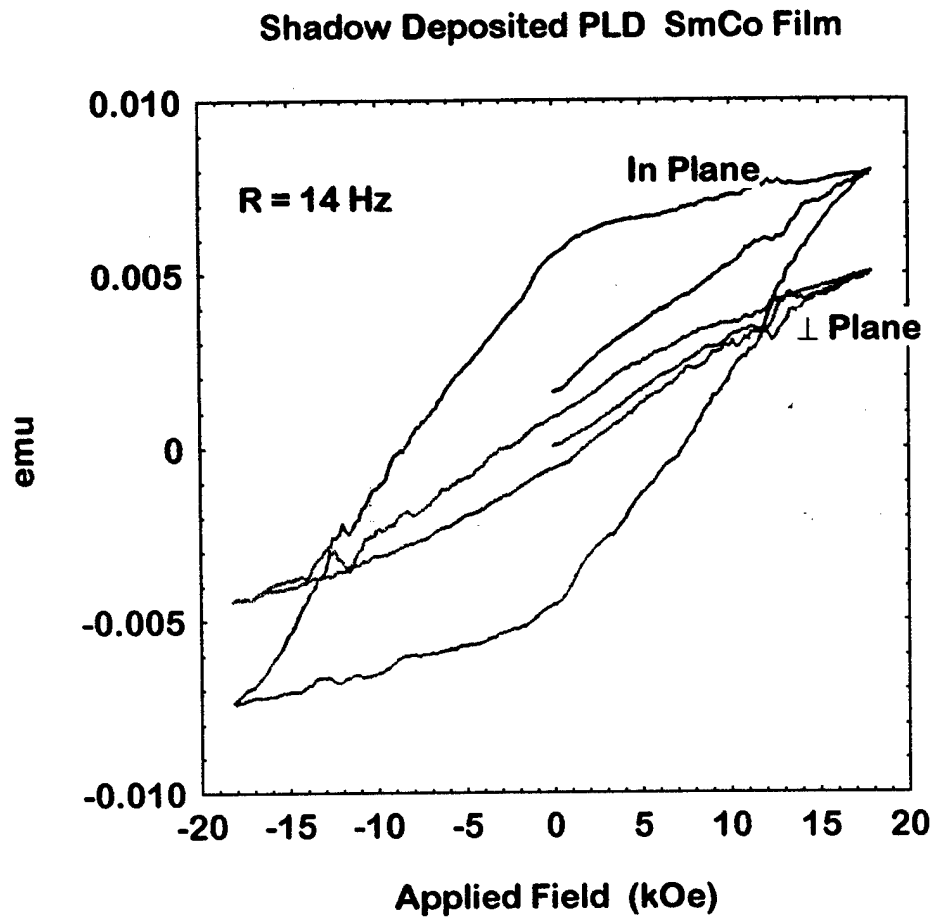


Fig. 8. Room temperature hysteresis loops for a shadow deposited PLD film made from  $\text{SmCo}_5$  target at  $375^\circ\text{C}$  on to alumina substrate, pressure 100 mTorr Ar, pulse rate 14 Hz, are shown. The key items to note are that the intrinsic coercivity is 9.7 kOe and that the loop shape is smooth.

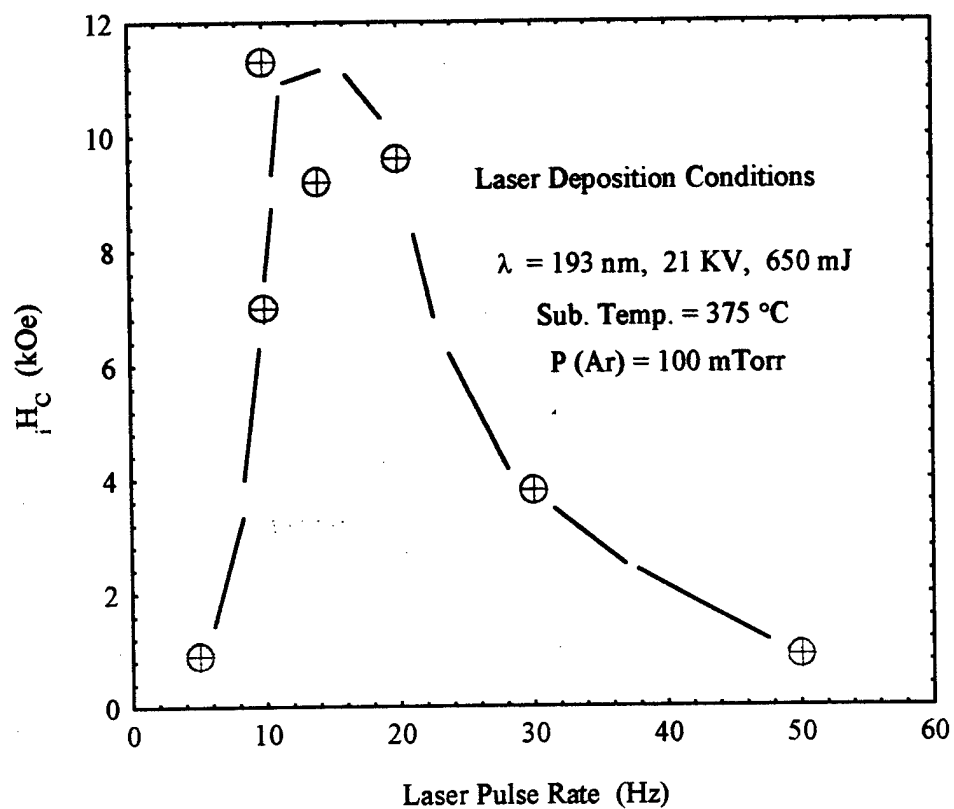


Fig. 9. The coercivity  $H_c$  versus laser pulse rate is shown for a series of films shadow deposited from  $\text{SmCo}_5$  bulk targets using  $\lambda = 193 \text{ nm}$ , T substrate = 375 °C, and P = 100 mTorr Ar. The values shown were measured at room temperature on as deposited films.

**C. LIST OF MANUSCRIPTS PUBLISHED UNDER ARO SPONSORSHIP DURING THIS GRANT, INCLUDING JOURNAL REFERENCE:**

1. A. Navarathna, P. Samarasekara, H. Hegde, R. Rani, and F.J. Cadieu, "Nitriding Studies of Aligned High Anisotropy  $\text{ThMn}_{12}$ -type  $\text{NdFe}_{11}\text{Co}_{1-y}\text{Mo}_y\text{N}$  Film Samples (Abstract)", J. Appl. Phys. **76**, 6068 (1994).
2. F. J. Cadieu, Recent Advances in Pseudobinary Iron Based Permanent Magnets, International Materials Reviews **40**, 137 (1995).
3. X. R. Qian, H. Hegde, and F. J. Cadieu, Aligned High Magnetization  $\text{Nd}(\text{Fe},\text{Co},\text{Mo})_{12}$  Films Nitrided By N Ion Implantation, J. Appl. Phys. **79**, 4614 (1996).
4. H. Hegde, X. R. Qian, Jong-Guk Ahn, and F. J. Cadieu, High Temperature Magnetic Properties of  $\text{TbCu}_7$ -type  $\text{SmCo}$  Based Films, J. Appl. Phys. **79**, 5961 (1996).
5. P. Samarasekara, R. Rani, F. J. Cadieu, and S. A. Shaheen, Variable Texture  $\text{NiOFe}_2\text{O}_3$  Ferrite Films Prepared By Pulsed Laser Deposition, J. Appl. Phys. **79**, 5425 (1996).
6. F. J. Cadieu, High Energy Product Permanent Magnet Films, Invited Paper, in Magnetic Materials, Processes, and Devices IV, L. T. Romankiw and D. A. Herman, Jr., Editors, PV 95-18, p. 319-335, The Electrochemical Society Proceedings Series, Pennington, NJ (1996).
7. M. Levy, R. M. Osgood, Jr., H. Hegde, F. J. Cadieu, R. Wolfe, and V. J. Fratello, Integrated Optical Isolators With Sputter-Deposited Thin-Film Magnets, Photonics Technology Letters **8**, 903 (1996).
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1. F. J. Cadieu, Recent Advances in Pseudobinary Iron Based Permanent Magnets, International Materials Reviews **40**, 137 (1995).
2. F. J. Cadieu, High Energy Product Permanent Magnet Films, Invited Paper, in Magnetic Materials, Processes, and Devices IV, L. T. Romankiw and D. A. Herman, Jr., Editors, PV 95-18, p. 319-335, The Electrochemical Society Proceedings Series, Pennington, NJ (1996).

#### **D. SCIENTIFIC PERSONNEL SUPPORTED BY THIS PROJECT**

- a. Professor Fred J. Cadieu, Principal Investigator
- b. Dr. Hari Hegde, Post Doctoral Research Associate
- c. Dr. X. R. Qian, Visiting Scholar
- d. Mr. Anil Navarathna, Ph.D. Graduate Student
- e. Ms. Raj Rani, Ph.D. Graduate Student
- f. Mr. Kieran Tracy, Queens College CUNY Undergraduate Student
- g. Mr. Pubudu Samarasekara, one third support as Ph.D. Graduate Student
- h. Dr. Raj Rani, Post Doctoral Research Associate
- i. Mr. W. Mendoza, Ph.D. Graduate Student, Florida State University
- j. Mr. Li Chen, Ph.D. Graduate Student, CUNY
- k. Mr. Theodore Theodoropoulous, Queens College CUNY Undergraduate Student
- l. Mr. Charles F. Cadieu, High School Student at Great Neck North High School, Great Neck, NY. This student was working on his science fair projects as a part of this grant.

#### **E. DEGREES AWARDED DURING THIS GRANT**

1. Ms. Raj Rani completed her Ph.D. thesis requirements and received the Ph.D. degree from the City University of New York, January 1995.
2. Mr. Anil Navarathna completed his Ph.D. thesis requirements and received the Ph.D. degree from the City University of New York, May 1995.
3. Mr. Pubudu Samarasekara completed his Ph.D. thesis requirements and received the Ph.D. degree from the City University of New York, January 1996.
4. Mr. Kieran Tracy completed B.A. Degree, Queens College CUNY, May 1995.

#### **5. Report of Inventions (By Title Only)**

None ascribed to ARO sponsorship.